

Ionizing Radiation Division	49010C	IRD-P-11
CALIBRATION IRRADIATIONS OF CUSTOMER SUPPLIED DOSIMETERS WITH ^{60}Co GAMMA RAYS		

Purpose

The purpose of this procedure is to describe the setup, measurement and reporting procedures for irradiation of customer supplied dosimeters.

Scope

This procedure covers the irradiation of passive dosimeters in the NIST ^{60}Co Gammacell irradiators. The irradiator dose rates are comparable to those used in the sterilization and radiation curing industries. Customer-supplied dosimeters are irradiated at prescribed temperatures and returned to the customer with an absorbed-dose irradiation certificate.

Definitions

Absorbed dose to water – the energy absorbed from ionizing radiation per unit mass of water: $1 \text{ J/kg} = 1 \text{ Gy}$.

5-hole cup – A polystyrene cylinder with 5 equidistant holes. Used for electronic equilibrium build-up material when irradiating perspex or ampoule dosimeters

Equipment

Essential Equipment	Calibration Method	Calibration Frequency
Vertical Beam Cobalt-60 Gamma-Ray Source	Water Calorimeter	Determined by control charts
MDS Nordion Gammacell 45	Comparison to Vertical Beam Source	Annual
MDS Nordion Gammacell 232	Comparison to Vertical Beam Source	Annual
MDS Nordion Gammacell 207	Comparison to Vertical Beam Source	Annual
Platinum Thermometer	External Service	Annual
Type-T Thermocouple	Comparison to Platinum Thermometer	Determined by control charts

Health & Safety Precautions

Radiation safety

Rooms containing ^{60}Co sources have been designated as High Radiation Areas. Specific requirements for entry and exit from the rooms are contained in these procedures and are posted on entry doors and walls. Radiation safety and training services are provided by the NIST Health Physics Office.

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Procedures[†]

Receive/Store Dosimeters

1. Upon receipt of shipment, inspect for any damage. The shipping package should be examined as well as the contents. Dosimeter packaging should be checked for damage to seals (if applicable). If the dosimeters are unsealed inspect them for any damage (i.e., cuts, breaks, scratches). If damage has occurred, follow Guide IRD-G07. In all instances, if there are any signs of damage, notify the customer.
2. In the Calibration Log Book, assign the next available HD number. Next to that, record the customer name, city, state, contact person, purchase order number, and date received.
3. Record the HD number on the Purchase order, then enter the data into the ISSC to generate the official test folder number.
4. Store the dosimeters in room B140 while waiting for irradiation. If the dosimeters are unpackaged radiochromic films, protect them from stray light, but leave them open to the 50 % relative humidity atmosphere.

Initiate Irradiation Data Record

1. Choose the appropriate geometry for the given dosimeters.
 - a. Perspex dosimeters go in the 5-hole cup
 - b. Alanine pellet dosimeters go in the single-hole vial
 - c. Pre-packaged films go in the film-block geometry with the 37mm square x 5mm polystyrene slabs
 - d. Unpackaged films go into the polystyrene film blocks with the machined center cavity.
 - e. Liquid ampoules go in the 5-hole cup.
2. Refer to the Irradiation Facilities Record Book to get the dose rate for the given irradiator and geometry.
3. Open the excel spreadsheet databook template and enter the appropriate header information, including the above mentioned annual dose rate.
4. Before each irradiation, enter an approximate value into the “start time” cell so the embedded function can calculate the decay, dose rate, and irradiation time.
5. Print a blank copy of the form “Timing Worksheet”, which should be a separate sheet within the excel file. This will be used to keep a handwritten record of the irradiation times.

Prepare Dosimeters for Irradiation

Perspex dosimeters:

1. Roll each dosimeter lengthwise and slip it into one of the 5 holes of the cup.
2. Choose an appropriate length polystyrene stem according to the printed table in

[†] The mention of commercial products throughout this paper does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that products identified are necessarily the best available for this purpose.

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the Irradiation Facilities Record Book.

3. Screw an aluminum base onto the stem.
4. Place 2 alanine pellets of the current QC batch into the top of the stem.
5. Place the 5-hole cup over the stem.

Alanine dosimeters:

1. Fill a single-hole vial with pellets. Depending on the pellet height, it will accept 3 or 4 pellets.
2. Choose an appropriate length polystyrene stem according to the printed table in the Irradiation Facilities Record Book.
3. Screw an aluminum base onto the stem.
4. Place 2 alanine pellets of the current QC batch into the top of the stem.
5. With a foam collar on the stem, place the single-hole vial into the collar to hold it atop the stem.

Film dosimeters:

1. Choose an appropriate length polystyrene stem according to the printed table in the Irradiation Facilities Record Book.
2. Screw an aluminum base onto the stem.
3. Place 2 alanine pellets of the current QC batch into the top of the stem.
4. Using electrical tape, secure the film block into an upright position atop the stem.

Control Sample Temperature

Samples are normally irradiated at room temperature, 22-25°C. GC45 does not require cooling to maintain ambient temperature, but to maintain that temperature in GC232 or GC207 requires blowing compressed air into the sample chamber. This is done by opening the valve of the flow meter mounted on the side of the appropriate Gammacell. Temperature is monitored with a type-T thermocouple placed inside the sample chamber. The type-T thermocouple is calibrated against the high-precision platinum thermometer in the Gammacell sample chamber (in the up position) over a temperature range that corresponds to service irradiations. The operational status of the thermocouple is monitored by periodic checks and control charts. Thermocouples that do not perform within the control limits are replaced.

On the computer, start the temperature recording program.

Active cooling with the turbojet air chiller is available for GC232 and GC207 to maintain sample temperatures down to -65°C. To operate the turbojet air chiller:

1. Turn the system on. Allow 5 minutes for system to get fully operational.
2. Switch turbojet from idle to manual mode and change setpoint to adjust sample temperature.
3. Monitor fluctuations in sample temperature and adjust turbojet setpoint when necessary to maintain the desired sample temperature.
4. When finished with the turbojet, put the system back to idle mode, start the defrost cycle, and switch off.

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Operate Gammacell

1. Place dosimeter assembly into the sample chamber.
2. Set timer to desired irradiation time, as calculated by the spreadsheet.
3. Press the “DOWN”(GC45) or “CYCLE START”(GC232) or “START”(GC207) button to begin the irradiation.
4. When the chamber arrives at the down position, note the actual clock time, write it on the timing worksheet, and enter it into the databook spreadsheet.
5. When the timer finishes and sample chamber comes up, remove the dosimeters.

Record Irradiation Data

1. After each irradiation, use the computer program to calculate the average temperature during irradiation. Enter the value into the databook spreadsheet.
2. When all irradiations are finished, save the file, print the spreadsheet and paste it into the High-Dose Irradiations databook. Store the timing worksheet with the other test folder documents.

Return Dosimeters

After all irradiations are completed, ship the dosimeters back to the customer. Package the dosimeters appropriately to avoid any damage during transit. Include a copy of the databook record. Also include a letter explaining that the given copy is unofficial and that the official record will be the formal certificate, which will be mailed later.

Analyze Quality Control

Measure the QC pellets as described in Section 4 of Procedure IRD-P-12. Prior to dose assessment, apply the correction factor to the response of the QC pellets to adjust for the different location within the geometry compared to the customer samples. Control charts of QC measurements are maintained to determine acceptance levels. If the measured dose is within control limits, the quality check is successful. If the measured dose is beyond the control limits, halt all irradiations and investigate to determine the source of the discrepancy. If the discrepancy can not be determined, the irradiations must be repeated.

Issue certificate and input ISSC to close folder

Once the quality check is successful, write a report titled “Absorbed-Dose Irradiation Certificate.” An example is shown in Appendix A. Route the report for the required signatures. Make two copies of the completed report before mailing the original to the customer. Note the mailed date on one of the copies. Log into the ISSC and close out the test and folder (reference ISSC quality manual).

Determination of uncertainties

The basis for the determination of uncertainties associated with high-dose irradiations is the Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results [1]. The purpose of this section is to explain the

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derivation of the various components of uncertainty for absorbed-dose certification. The values for the uncertainty components are listed in Appendix B.

Water Calorimetry: uncertainty from realization of the Gy [2].

Source Ratio Data: uncertainty from source dose-rate transfer (water calorimetry rate to high-dose calibration source rate) through ratio measurements.

Field Uniformity: radiation field uniformity within a dosimeter volume.

Timer: uncertainty of timer readout relative to shortest irradiation time interval.

Decay Correction: half-life correction factor uncertainty.

Traceability

The SI unit of absorbed dose is the Gray (Gy). For this service, the Gray is realized through water calorimetry measurements in the Vertical Beam Cobalt-60 Gamma-Ray Source. These measurements are transferred to the three GammaCell calibration sources by source-rate ratio measurements using alanine dosimetry. These transfer measurement protocols are described in NIST SP250-44 (See: <http://ts.nist.gov/ts/htdocs/230/233/calibrations/Publications/series-pdf/SP250-44.pdf>) [3].

Approximately annually, dosimetry comparisons are performed with the high-dose calibration facility of the National Physical Laboratory of the United Kingdom. Dosimeters from each facility are exchanged, measured, and the results compared. Participation in larger international comparisons occurs when appropriate. These data are summarized in the High-Dose International Comparisons Databook.

References

1. NIST Technical Note 1297, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results.
2. Domen, S.R., A sealed water calorimeter for measuring absorbed dose, J. Res. Natl. Inst. Stand. Technol., 99, pp. 121 – 141, 1994.
3. Radiation Processing Dosimetry Calibration Services and Measurement Assurance Program, Humphreys, J.C., Puhl, J.M., Seltzer, S.M., McLaughlin, W.L., Desrosiers, M.F., Bensen, D.L., Walker, M.L. 1998 NIST Special Publication 250-44.

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Records

Record	Contents/Purpose	Location
Calibration Log Book	Login all tests to obtain test folder number	C217
High-Dose Irradiations Databook	Records all dosimeter calibrations	C217
Irradiation Facilities Record Book	Records dose rates for irradiation geometries and instructions	B140
High-Dose International Comparisons Databook	Interlaboratory measurement comparisons data summaries	C217
Internal Calibrations	Source ratio measurements and data analysis	C209
GammaCell User Log Books	Cobalt-60 irradiator logs	B140

Filing and Retention

All paper copies of customer files are stored in the test folder for that service. All customer-related electronic files are stored either on password-protected calibration-staff desktops or in the “High Dose” folder on the shared network drive.

The IRD Quality Manager shall maintain the original and all past versions of this IRD Procedure. Copies of the current revision of this Procedure shall be placed in controlled Quality Manuals. Electronic copies of this Procedure are uncontrolled versions.

All deleted Procedures (including old revisions) shall be maintained by the IRD Quality Manager. All old revisions shall be maintained until such time as it is decided to delete the Procedure. Once the decision has been made to delete the Procedure, only the last revision shall be maintained by the IRD Quality Manager.

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Appendix A

National Institute of Standards and Technology

ABSORBED-DOSE IRRADIATION CERTIFICATE

Dosimeter
Film Dosimeters

Customer
Steel Curtain Irradiators, Inc.
00 Points Road
Pittsburg, PA 18888

ATTN: T. Bradshaw
Reference: PO# 2004-4444

Irradiation performed by James M. Puhl

Reviewed by Marc F. Desrosiers

Approved by
Stephen M. Seltzer, Leader
Radiation Interactions and Dosimetry Group

For the Director
National Institute of Standards and Technology
by
Lisa R. Karam, Acting Chief
Ionizing Radiation Division
Physics Laboratory

Information on technical aspects of this certificate may be obtained from James Puhl, NIST, 100 Bureau Drive
Stop 8460, Gaithersburg, MD 20899, 301-975-5581

*Report format revised May 30, 2003. The meaning of the data is identical to that previously transmitted as
"Report of Calibration."*

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Steel Curtain Irradiators supplied radiochromic film dosimeters in pre-sealed foil pouches. The dosimeters were irradiated using gamma radiation from a calibrated ^{60}Co irradiator, the NIST Gammacell 220-207. During irradiation, the dosimeter pouches were held in a polystyrene block assembly with a wall thickness of 5 mm. The dosimeters for sub-zero temperatures were held inside the stainless-steel dewar. The dates of irradiation, values of dose rate, absorbed dose, and mean irradiation temperature were as follows:

Dosimeter Identification	Date of Irradiation	Dose Rate (kGy/h)	Irradiation Temp. °C	Absorbed Dose kGy(H ₂ O)
1	May 20, 2003	18.55	23.5	3.800
2	May 20, 2003	18.55	23.8	5.500
3	May 20, 2003	18.55	24.6	12.00
4	May 20, 2003	18.55	25.2	13.00
5	May 23, 2003	18.53	24.5	22.00
6	May 21, 2003	18.54	24.4	22.00
7	May 21, 2003	18.54	24.5	24.00
8	May 23, 2003	18.53	24.6	33.00
9	May 21, 2003	18.54	24.9	34.00
10	May 21, 2003	18.54	24.3	42.00
11	May 21, 2003	18.54	24.3	44.00

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UNCERTAINTIES AND RELATED FACTORS IN HIGH-DOSE IRRADIATIONS

High-Dose Irradiations in Standard Geometries using the NIST ^{60}Co Gammacell 220-207

(Expanded uncertainty: $\pm 1.3\%$ at a 95 % confidence level)

The high-dose irradiations at NIST involve the administration of ^{60}Co gamma radiation under environmentally controlled conditions. The dose values are based on standard water calorimeter measurements and EPR/Alanine dosimetry, which are corrected by certain modifying factors (such as the geometry attenuation factor and source decay factor).

The uncertainty cited above is pertinent to absorbed dose in water in calibrated geometries. A detailed list of the various sources of uncertainty and estimates of the magnitude of those uncertainties that make up the overall uncertainty given above may be obtained by requesting such information from NIST. The uncertainties are divided into two types: A and B. Type A uncertainties are those evaluated by statistical methods, often associated with random effects. Type B uncertainties are those evaluated by other means, often associated with systematic effects.

Type A Uncertainties

The combined standard uncertainty evaluated by statistical methods is $\pm 0.25\%$ at an approximate level of confidence of 68 %.

Type B Uncertainties

The combined standard uncertainty based on scientific judgment is estimated to be $\pm 0.55\%$ at an approximate level of confidence of 68 %.

Expanded Uncertainty

The type A and type B uncertainties have been combined in quadrature (the square root of the sum of the squares) and multiplied by a coverage t-factor of 2.16 to yield an expanded uncertainty of $\pm 1.3\%$ at an approximate level of confidence of 95 %.

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Appendix B

Uncertainties of Irradiator Geometries

MDS Nordion Gammacell 45

GC45 Calibration Single Hole Geometry Dose Rate			
d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC45/Pool Source Ratio Data	0.09	
7	Pool/B036 Source Ratio Data	0.17	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.25	0.55
effective d.f.	combined in quadrature		0.60
17	t-factor for 17 d.f. at 95.45 %		2.16
Expanded Uncertainty at 95.45 %			
conf.			1.3

GC45 Calibration Film Block Geometry Dose Rate			
d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC45/Pool Source Ratio Data	0.09	
7	Pool/B036 Source Ratio Data	0.17	
2	Geometry Correction Factor	0.14	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.29	0.55
effective d.f.	combined in quadrature		0.62
16	t-factor for 16 d.f. at 95.45 %		2.17
Expanded Uncertainty at 95.45 %			
conf.			1.3

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GC45 Calibration Perspex Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC45/Pool Source Ratio Data	0.09	
7	Pool/B036 Source Ratio Data	0.17	
2	Geometry Correction Factor	0.03	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.25	0.55
effective d.f.	combined in quadrature		0.60
17	t-factor for 17 d.f. at 95.45 %		2.16
	Expanded Uncertainty at 95.45 % conf.		1.3

GC45 Calibration Alanine Ampoule Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC45/Pool Source Ratio Data	0.09	
7	Pool/B036 Source Ratio Data	0.17	
2	Geometry Correction Factor	0.11	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.27	0.55
effective d.f.	combined in quadrature		0.61
18	t-factor for 18 d.f. at 95.45 %		2.15
	Expanded Uncertainty at 95.45 % conf.		1.3

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MDS Nordion Gammacell 232

GC232 Calibration Single Hole Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC232/Pool Source Ratio Data	0.10	
7	Pool/B036 Source Ratio Data	0.17	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.25	0.55
effective d.f.	combined in quadrature		0.60
18	t-factor for 18 d.f. at 95.45 %		2.15
	Expanded Uncertainty at 95.45 % conf.		1.3

GC232 Calibration Film Block Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC232/Pool Source Ratio Data	0.10	
7	Pool/B036 Source Ratio Data	0.17	
2	Geometry Correction Factor	0.04	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.26	0.55
effective d.f.	combined in quadrature		0.61
18	t-factor for 18 d.f. at 95.45 %		2.15
	Expanded Uncertainty at 95.45 % conf.		1.3

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GC232 Calibration Perspex Geometry Dose Rate			
d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC232/Pool Source Ratio Data	0.10	
7	Pool/B036 Source Ratio Data	0.17	
2	Geometry Correction Factor	0.11	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.28	0.55
effective d.f.	combined in quadrature		0.61
19	t-factor for 19 d.f. at 95.45 %		2.14
	Expanded Uncertainty at 95.45 % conf.		1.3

GC232 Calibration Alanine Ampoule Geometry Dose Rate			
d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
11	GC232/Pool Source Ratio Data	0.10	
7	Pool/B036 Source Ratio Data	0.17	
2	Geometry Correction Factor	0.07	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.26	0.55
effective d.f.	combined in quadrature		0.61
19	t-factor for 19 d.f. at 95.45 %		2.14
	Expanded Uncertainty at 95.45 % conf.		1.3

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MDS Nordion Gammacell 207

GC207 Calibration Single Hole Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
7	GC207/Pool Source Ratio Data	0.08	
7	Pool/B036 Source Ratio Data	0.17	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.25	0.55
effective d.f.	combined in quadrature		0.60
16	t-factor for 16 d.f. at 95.45 %		2.17
	Expanded Uncertainty at 95.45 % conf.		1.3

GC207 Calibration Film Block Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
7	GC207/Pool Source Ratio Data	0.08	
7	Pool/B036 Source Ratio Data	0.17	
3	Geometry Correction Factor	0.05	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.25	0.55
effective d.f.	combined in quadrature		0.60
17	t-factor for 17 d.f. at 95.45 %		2.16
	Expanded Uncertainty at 95.45 % conf.		1.3

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GC207 Calibration Perspex Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
7	GC207/Pool Source Ratio Data	0.08	
7	Pool/B036 Source Ratio Data	0.17	
3	Geometry Correction Factor	0.10	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.27	0.55
effective d.f.	combined in quadrature		0.61
19	t-factor for 19 d.f. at 95.45 %		2.14
	Expanded Uncertainty at 95.45 % conf.		1.3

GC207 Calibration Alanine Ampoule Geometry Dose Rate

d.f.	Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in vertical beam	0.16	0.51
7	GC207/Pool Source Ratio Data	0.08	
7	Pool/B036 Source Ratio Data	0.17	
3	Geometry Correction Factor	0.07	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	⁶⁰ Co Decay Correction		0.02
	sqrt(sum)	0.26	0.55
effective d.f.	combined in quadrature		0.61
18	t-factor for 18 d.f. at 95.45 %		2.15
	Expanded Uncertainty at 95.45 % conf.		1.3

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